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TITLE: Prepn. of sugar cane wax - by chipping surface of cane and extracting the chipped cane obtd with an organic solvent

PATENT-ASSIGNEE: NIPPON PETROCHEMICALS CO LTD[NIPE]

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APPLICATION-DATA:

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ABSTRACTED-PUB-NO: JP06200288A

BASIC-ABSTRACT: In a new prepn. of sugar cane wax, the surface portion of up to 10% of the thickness in the radial direction of the stem is chipped from the sugar cane; and the chipped surface portion is used as a raw material. Pref. the sugar cane wax is extracted from the chipped surface portion.

The extracting solvent is e.g. hexane, heptane, ethanol, benzene, diethyl ether, methyl ethyl ketone and/or chloroform.

USE/ADVANTAGE - The easy method provides high-quality sugar cane wax with a single, high m.pt. contg. practically no impurities.

In an example, the surface portion of 1.5% of the dia. of stem (dia., 41 mm) of sugar cane was chipped. The chipped surface portion was dried in a vacuum drier and extracted with n-heptane at its b.pt. for 6 hr, with refluxing, to obtain wax soln. The soln. was distilled to remove the solvent to obtain light-yellow sugar cane wax. No visual impurities were observed in the wax; and it was confirmed by gas chromatography that the wax contained no undesirable ingredients. The wax obtd. showed a single sharp peak at 81.0 deg. C in DSC.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS:

PREPARATION SUGAR CANE WAX CHIP SURFACE CANE EXTRACT CHIP CANE OBTAIN ORGANIC SOLVENT

DERWENT-CLASS: D23

CPI-CODES: D06-A; D10-B;

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CLIPPEDIMAGE= JP406200288A

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TITLE: PRODUCTION OF SUGAR CANE WAX

PUBN-DATE: July 19, 1994

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APPL-NO: JP04360206

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APPL-DATE: December 28, 1992

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US-CL-CURRENT: 554/20

ABSTRACT:

PURPOSE: To easily produce a high-quality sugar cane wax having a single high melting point and being substantially free from impurities by using as a raw material an outer surface layer of a sugar cane stalk which layer has a specific thickness.

CONSTITUTION: An outer surface layer of a sugar cane stalk is peeled off to a thickness which is 10% or below, preferably 5% or below, of the stalk diameter (usually 20-100mm). For producing a sugar cane wax from the surface layer, a known method may be used without particular limitation. For example, an organic solvent (e.g. hexane) is used to extract the wax at a temp. not lower than the melting point of the wax to be obtained (e.g. 75-85deg;C) for usually

0.1-10hr. Supercritical extraction using carbon dioxide, etc., is also usable.  
For example, the sugar cane surface layer is dried in a vacuum and put in a pressure vessel and the vessel is filled with carbon dioxide to conduct extraction at  $280\text{kg/cm}^2$  and  $80^\circ\text{C}$ , following which the contents are discharged from the vessel into a receiver under ordinary pressure to obtain the desired wax in powdery form.

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## (54) PRODUCTION OF SUGAR CANE WAX

### (57)Abstract:

**PURPOSE:** To easily produce a high-quality sugar cane wax having a single high melting point and being substantially free from impurities by using as a raw material an outer surface layer of a sugar cane stalk which layer has a specific thickness.

**CONSTITUTION:** An outer surface layer of a sugar cane stalk is peeled off to a thickness which is 10% or below, preferably 5% or below, of the stalk diameter (usually 20-100mm). For producing a sugar cane wax from the surface layer, a known method may be used without particular limitation. For example, an organic solvent (e.g. hexane) is used to extract the wax at a temp. not lower than the melting point of the wax to be obtained (e.g. 75-85°C) for usually 0.1-10hr. Supercritical extraction using carbon dioxide, etc., is also usable. For example, the sugar cane surface layer is dried in a vacuum and put in a pressure vessel and the vessel is filled with carbon dioxide to conduct extraction at 280kg/cm<sup>2</sup> and 80°C, following which the contents are discharged from the vessel into a receiver under ordinary pressure to obtain the desired wax in powdery form.

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]**

[0001]

[Field of the Invention] this invention relates to the manufacture technique of a new cane wax.

[0002]

[Description of the Prior Art] The animals-and-plants system wax which has the characteristic feature which is not in a mineral oil system wax is extracted by various technique from various raw materials, and is used in various industrial fields taking advantage of each characteristic feature. Use is proposed also for the cane wax contained in the cane as those one. By the way, conventionally, sugar presses out a sap from the stalk of a cane and is manufactured from the drawing juice. That is, the whole stalk of a cane is ground suitably, this is squeezed, and the sap in a stalk is pressed out. The sap of a cane is manufactured in large quantities for the sugar production, and the amount of [ of a cane ] wax is floating in the state of an emulsion in the sap. Therefore, the method of extracting a part for a wax out of the sap pressed out from the cane as the manufacture technique of a cane wax is performed.

[0003]

[Problem(s) to be Solved by the Invention] However, when this invention persons examined the property of the cane wax manufactured from the drawing juice of the whole stalk of a cane as mentioned above, the quality was not what may not necessarily be satisfied. That is, there is what two or more peaks which show the melting point by DSC (differential scanning calorimetric analysis) may exist, and shows the peak of the low melting point in it in the wax manufactured by the above-mentioned technique. It expresses that shows that a wax component is not single, including an impurity, and is [ that two or more peaks which show the melting point exist ] wide collectively, and synchysis nature is not sharp. [ of a melting-temperature domain ] As a wax, these do not spoil the physical properties generally demanded and are not desirable. Therefore, in order to obtain the cane wax as a product, it is necessary to remove these impurities. However, since processes, such as filtration, adsorption, or solvent refining, are required for elimination of an impurity, these must be applied in addition to an extraction of a wax, and a cost cannot but go up remarkably.

[0004]

[Means for Solving the Problem] In case they extract a part for a wax from a cane in view of the above situation, as a head end process, this invention persons shave off the stalk front face of a cane, by using the extracted stalk epidermis fraction as a raw material, find out that the cane wax which does not contain an impurity substantially can be manufactured, and complete this invention. That is, the manufacture technique of the cane wax characterized by this invention using as a raw material the epidermis fraction which shaved off and extracted the stalk epidermis fraction by 10% or less of the thickness of a diameter toward the core from the stalk front face of a cane is offered. According to the manufacture technique of the cane wax of this invention, there are very few contents of an impurity, or the cane wax which does not contain an impurity substantially can be obtained, it becomes unnecessary in practice to refine, in case it is commercial production, and a manufacturing cost can be improved remarkably.

[0005] In the manufacture technique of the cane wax of this invention, the thing of the stalk diameter of a cane for which a stalk epidermis fraction is preferably shaved [ front face / stalk / of a cane ] by 5% or less of thickness 10% or less toward a core is important. It is not desirable, in order that mixing of a component to which the physical properties of a wax are reduced may increase, while the extraction efficiency of a wax will fall, if the thickness exceeding 10% is shaved off. That is, when the epidermis fraction of the thickness exceeding 10% is included, two or more peaks which show the melting point by DSC to the obtained wax are seen, or the peak of the low melting point accepts. The technique of this invention does not receive a limit in growth of a cane, the degree of maturation, or its modality. Usually, the stalk diameter of a cane is about 20-100mm.

[0006] In the technique of this invention, although the stalk epidermis fraction of predetermined thickness is shaved

[ front face / stalk / of a cane ] toward the orientation of a core as mentioned above, especially this operating instruction is not limited. For example, in case you may carry out by operation by a man's hand and a lot of processing is performed, you may carry out by operation by the machine. What canna, a grinder, etc. which a limit does not have in a modality if it can shave off in fixed thickness, for example, are used for wood working etc. as a machine processed in large quantities write, and is similar to a machine or these is used. Moreover, as equipment which writes automatically and performs \*\*\*, it can tear apart with a conveyer which is shown in JP,55-2280,B, and a blade and the complicated equipment which wrote and combined the roll of business etc. can also be used, and let all the epidermis fractions of the cane obtained by these machines and equipments be the objects of a manufacture of this invention.

[0007] In order to manufacture a cane wax from the stalk epidermis of the shaved-off cane, well-known technique can be used conventionally and it is not limited especially. For example, the shaved-off cane stalk epidermis is squeezed and a wax is extracted from the sap as obtained drawing juice. Or flocculants, such as a calcium hydroxide and a tricalcium phosphate, can be added further, a wax emulsion can be settled, and a cane wax can also be manufactured by the technique of extracting this sedimentation after a collection. However, the technique of setting the stalk epidermis of the shaved-off cane as the object of a direct extraction is economical, and since what was excellent also in the quality is obtained, it is desirable.

[0008] The extract operation using a suitable extractant can be mentioned as a typical thing of the technique of setting the stalk epidermis of a cane as the object of a direct extraction. As an extractant, the well-known organic solvent for an extraction is illustrated conventionally. For example, chlorinated hydrocarbons, such as ketones, such as ether, such as aromatic hydrocarbons, such as alcohols, such as aliphatic hydrocarbon, such as a hexane and a heptane, and ethanol, propanol, or benzene, and a xylene, and diethylether, and a methyl ethyl ketone, and chloroform, are mentioned. Since water lives together when setting the shaved-off cane stalk epidermis as the object of a direct extraction, or in setting cane stalk epidermis as the object of an extraction of the sap squeezed and obtained, it is desirable to make the organic solvent of a canal system into an extraction solvent. When the organic solvent of a chlorinated-hydrocarbon system or the organic solvent of an aromatic-hydrocarbon system was used as an organic solvent of a canal system and these remain in a wax, a problem is produced from the point of the safety of a product wax. Therefore, it is desirable to make the saturated hydrocarbons containing aliphatic hydrocarbon, such as a hexane and a heptane, into an extraction solvent.

[0009] Extract operation by the organic solvent is performed within the operating temperature beyond the melting point of the wax obtained, for example, a 75-85-degree C temperature requirement, and extraction time is usually 0.1 - 10 hours.

[0010] In addition, the supercritical-extraction technique using the technique of extracting by extractants other than the organic solvent, for example, the method of warm temperature being under water bath and extracting a wax, carbon dioxide gas, etc. is employable. However, the technique of extracting from the point of economical efficiency or wax recovery luminous efficacy by the organic solvent is industrially advantageous. The cane wax made into the purpose can be suitably obtained easily by removing an extractant by distillation by well-known technique after an extraction.

[0011]

[Example] An example explains this invention in detail below.

The front face of the stalk (diameter of 41mm) of <example 1> Okinawa \*\*\*\*\* to diameter It shaved off toward the core by 1.5% of thickness, and the cane epidermis fraction was obtained. The wax was extracted, flowing back with the boiling point for 6 hours, after drying this by the vacuum dryer using n-heptane as a solvent. The obtained wax solution was distilled, the solvent was removed and the cane wax of light yellow was obtained. It was checked that impurities, such as dust, are not accepted in the obtained wax by viewing, and an unnecessary component except a wax part is not included as a result of analysis by the gas chromatograph. In the result of DSC, the obtained cane wax has a sharp peak single 81.0 degrees C, and it turns out that it shows sharp synchysis nature.

[0012] <Example 2> It is a cane diameter about the thickness to shave off. The cane wax was extracted like the above-mentioned example 1 except having considered as 0.5%. A component except a wax part was not detected as a result of gas-chromatograph analysis. Moreover, in the result of DSC, the sharp peak single 80.7 degrees C was seen.

[0013] It shaved [ front face / of the same cane as the <example 1 of comparison> example 1 ] by 20% of thickness toward the center, and extracted by the same technique as the above-mentioned example 1. As a result of carrying out gas-chromatograph analysis of the wax of the obtained light green, it turns out that components other than a wax part are contained 15%. Moreover, according to the result of DSC, it has a peak in three places, 49 degrees C, 67 degrees C, and 76 degrees C, and it turns out that synchysis nature is not sharp.

[0014]

[The example 2 of a comparison] The filter cake discharged by the raw sugar manufacturing process was dried by the

vacuum dryer, and extract operation was performed like the example 1. As a result of seeing some solid impurity in the wax of the obtained dark green and a gas chromatograph's analyzing this, it turns out that components other than a wax part are contained 37%. In the result of DSC, two peaks were shown in 47 degrees C and 66.5 degrees C, and it turns out that synchysis nature does not have the low sharp melting point, either.

[0015]

[Example 3] Using the grinder for wood working, the epidermis fraction of the cane stalk front face of an example 1 was shaved off so that it might be thin in 5% of a cane diameter, and the cane wax was extracted by the same technique as an example 1. A component except a wax part was not detected as a result of gas-chromatograph analysis. Moreover, the peak single 80.5 degrees C was seen in the result of DSC.

[0016]

[Example 4] Using the equipment which divides the stalk of a cane into an epidermis fraction, a continuous-glass-fiber fraction, and a main \*\*\*\*\* fraction automatically, the stalk epidermis fraction of the cane of an example 1 was shaved off by 1 - 3% of the thickness of the diameter of a cane, and the cane wax was extracted by the same technique as an example 1. A component except a wax was not detected as a result of gas-chromatograph analysis. Moreover, the peak single 80.1 degrees C was seen in the result of DSC.

[0017]

[Example 5] The front face of the stalk of the Okinawa \*\*\*\*\* of an example 1 to diameter Only 2.5% of thickness was shaved off toward the core, and the cane stalk epidermis fraction was obtained. After drying this by the vacuum dryer, it put in into the pressure-proof container, it was filled up with carbon dioxide gas, and extracted at pressure 2 and temperature of 80 degrees C of 280kg/cm. The wax extracted from the pressure-proof container with the carbon dioxide gas of a super-critical state was emitted into the receiver of an ordinary pressure, carbon dioxide gas was removed as a gas, and the powdered cane wax was obtained in light yellow. It was checked that impurities, such as dust, are not accepted in the obtained wax by viewing, and a component except a wax part is not included as a result of analysis by the gas chromatograph. In the result of DSC, the obtained cane wax has a sharp peak single 81.8 degrees C, and it turns out that it shows sharp synchysis nature.

[0018]

[Example 6] The front face of the stalk of the Okinawa \*\*\*\*\* of an example 1 to diameter Only 1.5% of thickness was shaved off toward the core, and the cane stalk epidermis fraction was obtained. It boils in a boiling water for 5 hours, and the wax layer which floats in the upper part is dipped up, keeping it warm, it put for 1.5 hours and the wax layer and the water layer were made to separate, after drying this by the vacuum dryer. This was cooled, the wax layer was solidified, moisture was removed, and the cane wax of light yellow was obtained. It was checked that the obtained wax does not contain a component except a wax part by viewing as a result of analysis [ impurity dust ] according [ do not accept and ] to a gas chromatograph. In the result of DSC, the obtained cane wax has a sharp peak single 79.8 degrees C, and it turns out that it shows sharp synchysis nature.

[0019]

[Effect of the Invention] According to the manufacture technique of this invention, the quality cane wax which has the single high melting point and which does not contain an impurity substantially can be manufactured easily.

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[Translation done.]